UNCLASSIFIED

AD 405 135

DEFENSE DOCUMENTATION CENTER

FOR

SCIENTIFIC AND TECHNICAL INFORMATION

CAMERON STATION, ALEXANDRIA, VIRGINIA

UNCLASSIFIED

63-3-5

ASD-TDR 7-946 (III)

May, 1963

DEVELOPMENT OF DIES FOR EXTRUSION OF COMPLEX SHAPES OF STEEL AND REFRACTORY ALLOYS

J. G. Hunt, J. L. Klein, A. M. White, and R. G. Jenkins

INTERIM TECHNICAL DOCUMENTARY PROGRESS REPORT
NO. ASD-TDR 7-946 (III)
1 January 1963 - 31 March 1963

Manufacturing Technology Laboratory
Aeronautical Systems Division
Air Force Systems Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

ASD Project No. 7-946
NMI Project No. 1597

Work continued on methods of fabricating and evaluating dies suitable for the high temperature extrusion of steel and refractory metals of complex shapes. Fabrication of ceramic and "intermetallic" dies by hot pressing and by melting and forging continued. Successful methods of producing tungsten fibers for reinforced dies were developed. A die support system based on the concept of hydrostatic support for the die casing has been developed and shown to be successful in preventing the cracking of brittle die nibs during extrusion.

Prepared under Contract AF33(657)-8798
Nuclear Metals, Inc., West Concord, Massachusetts

nuclear metals, inc.
concord, massachusetts

NOTICES

FOREWORD

This Interim Engineering Progress Report covers the work performed under Contract AF 33(657)-8798 from 1 January 1963 through 31 March 1963. It is published for technical information only and does not necessarily represent the recommendations, conclusions or approval of the Air Force.

This contract with Nuclear Metals, Inc., West Concord, Massachusetts, was initiated under ASD Project 7-946, "Development of Die Materials and Dies." It is being accomplished under the technical direction of T. S. Felker, Manufacturing Technology Laboratory, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

Messrs. J. G. Hunt, Research Ceramist, R. G. Jenkins, Chemical Metallurgist, and A. M. White, Group Leader, are the Nuclear Metals personnel most actively engaged in the program. Others who contribute to the work at Nuclear Metals are Mr. J. L. Klein, Vice President for Operations, Mr. P. Loewenstein, Department Director, and Dr. A. R. Kaufmann, Technical Director and Vice President. This report has been given the Nuclear Metals, Inc. internal report number NMI-9700.9.

The primary objective of the Air Force Manufacturing Methods Program is to develop on a timely basis manufacturing processes, techniques and equipment for use in economical production of USAF materials and components. The scope of this particular program is to develop new dies for use in hot-extruding refractory metals and steel. The dies to be investigated fall into three classes: ceramic-coated metallic materials, metal-fiber-reinforced ceramic materials, and refractory "intermetallic" materials.

Your comments are solicited on the potential utilization of the information contained herein as applied to your present or future production programs. Suggestions concerning additional Manufacturing Methods development required in this or other subjects will be appreciated.

Written by: J G Hunt

J. G. Hunt
Research Ceramist

PUBLICATION REVIEW:

Approved by: Paul Loewenstein

P. Loewenstein
Director, Mechanical Metallurgy Department

Approved by: J L Klein

J. L. Klein
Vice President for Operations

Approved by: A. Kaufmann

A. R. Kaufmann
Vice President and Technical Director

ASD-TDR 7-946 (III)
May, 1963

DEVELOPMENT OF DIES FOR EXTRUSION OF COMPLEX SHAPES OF STEEL AND REFRACTORY ALLOYS

J. G. Hunt, J. L. Klein, A. M. White, and R. G. Jenkins
Nuclear Metals, Inc.

Work continued on methods of fabricating and evaluating dies suitable for the high temperature extrusion of steel and refractory metals of complex shapes. Fabrication of ceramic and "intermetallic" dies by hot pressing and by melting and forging continued. Successful methods of producing tungsten fibers for reinforced dies were developed. A die support system based on the concept of hydrostatic support for the die casing has been developed and shown to be successful in preventing the cracking of brittle die nibs during extrusion.

In the third quarter (1 January 1963 through 31 March 1963), progress has been made in the following areas: (1) fabrication of "intermetallic" dies by both hot pressing and melting and forging; (2) obtaining "intermetallic" dies from outside sources; (3) finish grinding of round "intermetallic" dies; (4) establishment of feasibility of hot pressing Tee dies; (5) developing methods of making tungsten reinforcing fibers by rolling and swaging; (6) development of a method for supporting dies so that the pressure from the extrusion billet results in a compressive force on the die nib to prevent cracking; (7) establishing the value of refractory metals as promising substrates for coated dies; (8) modification of a high temperature furnace to provide a 2 to 3 second loading time for TZM billets. It has been established that the indenter test in its present state of development is not a satisfactory means of screening die materials, and further work with the indenter test will be held in abeyance.

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

I. INTRODUCTION

A. General

The objective of this program is to develop a die for the high temperature extrusion of complex structural shapes of steel and refractory metal alloys. Prescribed reference alloys are AISI-SAE Type 4340 steel (0.40 C, 1.75 Ni, 0.80 Cr, 0.25 Mo) and molybdenum alloy TZM (Mo - 0.5 Ti - 0.08 Zr). The reference cross-section of the metal to be extruded is a Tee having a flange approximately two inches wide and a stem approximately one inch deep; the maximum thickness of flange and stem are 1/16 inch for the steel and 1/4 inch for the TZM alloy.

Demonstration of success in meeting the program objective will be the extrusion of twenty or more feet of uniform Tee shape with minimum deterioration of the die. The extrusion equipment shall enable stressing the ram to 90 tsi, and furnaces shall be capable of heating the TZM billets to 3400°F (1870°C).

In the original program scope, three types of dies were to be investigated consecutively: ceramic-coated metals (Phase I), metal fiber-reinforced ceramics (Phase II), and refractory compounds and intermetallics (Phase III). As the work progressed the advantages of concurrent work on all three phases became apparent, and the program has been changed accordingly.

B. Review of Previous Work in Program

Work performed during the first quarter (July through September, 1962)[1] was concerned principally with planning the experimental program. After a review of extrusion facilities and reported materials properties, selection was made of candidate die materials, fabrication techniques, evaluation procedures, and die support systems.

For ceramic-coated metal substrate dies (Phase I), steel and/or refractory metals with zirconia-based coatings applied by plasma and flame spraying were chosen. For fiber-reinforced dies (Phase II), hot pressed zirconium silicate and silicon nitride, each reinforced with tungsten fibers, were chosen. The bases for selection of these materials for fiber-reinforced dies

were: (1) tungsten has a high elastic modulus, high strength over a wide temperature range, and is readily available in a form that can be converted into fibers; (2) zirconium silicate and silicon nitride do not react appreciably (if at all) with tungsten; and (3) the higher expansion coefficient of tungsten results in the desired residual compressive stresses in the ceramic matrix after cooling from the hot pressing operation used for making the dies. For massive refractory compound dies (Phase III), various refractory silicides, carbides, borides and nitrides, either self- or otherwise-bonded, and made by hot pressing or sintering, were selected.

Two procedures were selected for evaluation of die materials: (1) an indenter test, intended to be an economical and simple means of simulating extrusion conditions; and (2) actual extrusion through die materials that showed promise from the indenter test. Initially materials were to be fabricated and tested first as indenters, then as round dies, and finally as Tee dies. Each type of indenter specimen was to be tested at 2200°F (1200°C) with steel billets and then 3400°F (1870°C) with TZM billets.

Although external die supports are necessary for Tee dies (because of the dimensions of the Tee relative to the extrusion liner), internal supports were planned for round dies for convenience and economy. In addition, since a portion of even an external die usually extends into the extrusion container to prevent leakage of metal under high pressures during extrusion, development of supports for internal round dies was anticipated to assist in the development of supports for external Tee dies.

Work performed during the second quarter (October through December, 1962)[2] was concerned primarily with preliminary experiments to verify and delineate further the planning of the program. Preliminary fabrication trials resulted in successful hot pressing of metal-bonded oxides (tungsten powder plus Al_2O_3 and tungsten powder plus ThO_2). In extrusion tests of AISI 4340 steel through round dies, these metal-bonded oxide dies were superior to uncoated H21 steel dies. Hot pressing techniques were investigated for a number of ceramic and metal-ceramic dies, such as $MoSi_2$, B_4C, W-$MoSi_2$, W-ZrB_2, W-ZrC, W-WC, W-SiC, W-TiC-Al_2O_3, W-B_4C-Al_2O_3, WC-Al_2O_3, and others. About

one third of the hot pressings resulted in successful dies. Three types of die supports -- called "soft," "hard," and "isostatic" -- were tried, and none was completely successful.

II. CURRENT WORK

A. General

In the third quarter (January 1, 1963 through March 31, 1963), progress has been made in the following areas: (1) fabrication of "intermetallic" dies by both hot pressing and melting and forging; (2) obtaining "intermetallic" dies from outside sources; (3) finish grinding of round "intermetallic" dies; (4) establishment of feasibility of hot pressing Tee dies; (5) developing methods of making tungsten reinforcing fibers by rolling and swaging; (6) development of a method for supporting dies so that the pressure from the extrusion billet results in a compressive force on the die nib to prevent cracking; (7) establishing the value of refractory metals as promising substrates for coated dies; (8) modification of a high temperature furnace to provide a 2 to 3 second loading time for TZM billets. It has been established that the indenter test in its present state of development is not a satisfactory means of screening die materials, and further work with the indenter test will be held in abeyance.

B. Experiments and Results

1. Fabrication of Die Materials

With the use of equipment described previously,[1] a series of experimental materials was hot pressed into round nibs. Two nibs of the same material, one each with die lands of 0.400 and 0.750 inch diameter, were made for testing with 4340 steel and TZM alloy, respectively, as described in Table 1. Pressures of one tsi and pressing times of 10 minutes in graphite molds at various holding temperatures were used. Compositions were primarily oxide-bonded borides, carbides, and nitrides and tungsten-bonded nitrides and silicates. In a total of thirty hot pressed nibs,

TABLE 1

Fabrication Data for Experimental Hot Pressed Nibs

Pressing No.*	Composition, v/o	Pressing Temp.**		Density		Visual Appearance
		°C	°F	Absolute, g/cm^3	% of Theoretical	
41 A,B	TiB_2 - 20 Al_2O_3	1950	3540	4.40	100	Good, dense
42 A,B	TiB_2 - 20 ThO_2	2050	3720	5.6	>100	Good, dense
44 A,B	ZrB_2 - 20 Al_2O_3	1900	3450	5.55	99	Good, dense
45 A,B	ZrB_2 - 20 ZrO_2	1950	3540	--	--	Cracks, dense
43 A,B	ZrB_2 - 20 ThO_2	2000	3630	--	--	Cracks, dense
46 A,B	ZrC - 10 $MoSi_2$	2000	3630	6.0	91	Good, dense
48 A,B	ZrC - 2 Al_2O_3	2150	3900	6.28	92	Good, dense
49 A,B	ZrC - 2 TiO	1650	3000	--	--	Porous
53 A,B	$SiZrO_4$ - 20 W	1500	2730	6.8	92	Porous
55 A,B	$SiZrO_4$ - 20 W	1600	2910	7.0	93	Good, dense
52 A,B	Si_3N_4 - 20 W	1550	2820	--	--	Porous
54 A,B	Si_3N_4 - 20 W	1650	3000	--	--	Porous
50 A,B	Si_3N_4 - 5 MgO	1850	3360	3.1	98	Good, dense
51 A,B	Si_3N_4 - 5 MgO - 20 W	1650	3000	5.9	95	Good, dense

* Die A has a 0.400 inch inside diameter and die B a 0.750 inch ID; nib outside diameter was 2.00 inches, and nib lengths ranged from 1-1/4 inches to 1-3/4 inches.

** Nibs were pressed in graphite molds at temperature indicated for 10 minutes at a pressure of approximately one tsi.

eighteen were considered suitable for further processing, and the remainder unsuitable because of porosity or cracks.

Because of the extreme hardness and refractoriness reported for experimental alloys W - 5.5 $^{w}/o$ Ru and Ta - 6 $^{w}/o$ Ru (investigated in other programs under sponsorship of the Air Force[3] and the Atomic Energy Commission[4]), these compositions were also fabricated into dies. Elemental powders were blended, pressed into pellets, arc melted, canned in molybdenum, and upset forged in an "insulated" extrusion container at 3600°F (2000°C) and 100 tsi. Each "forging" was cut in half transversely, machined, and shrunk into a steel casing.

In addition to the nibs fabricated at NMI by hot pressing and alloying, as described above, others have been obtained from outside sources. A summary of ceramic and intermetallic nibs available or in process for experimental dies is given in Table 2. During the third quarter approximately fifteen nibs were ground to final size. These nibs will be shrunk into casings to make dies for trial extrusions in the fourth quarter.

A preliminary pressing of a Tee die made of W - 33 $^{v}/o$ ThO_2 was fabricated by hot pressing at a temperature of 3200°F (1750°C) and a pressure of approximately one tsi in an ATJ grade* graphite mold to investigate parameters of mold design for Tee shaped dies. A Tee with complex entry shape and good surface in the as-pressed condition was obtained, as shown in Fig. 1. No relief of the die land was attempted in this first pressing. A steel casing with 5-mil interference fit was successfully shrunk around the nib without causing cracking of the nib.

In order to fabricate tungsten fiber reinforced ceramics, experiments were carried out to develop a process for tungsten fiber manufacture. The selection of desirable lengths and fiber diameters was based on experiments described by Tinklepaugh,[5] who reported that the best results were obtained with 1/8 to 1/2 inch lengths in diameters less than 6 mils. Experiments at NMI were performed to produce material in this size range. Tungsten wool, a form of loosely matted wire scrap from commercial wire drawing manufacturing, was used as the starting material because it is more economical and

---

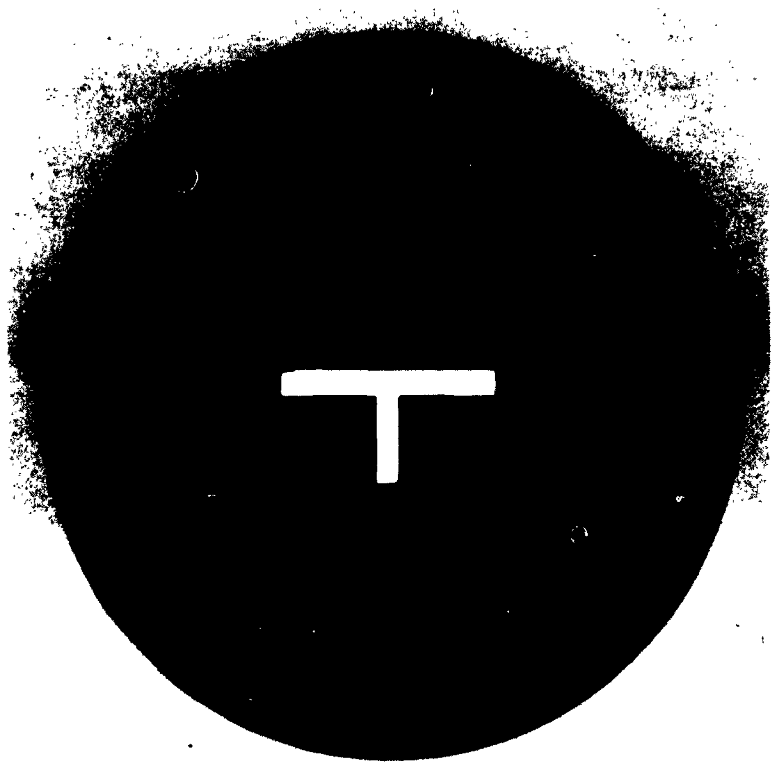
* National Carbon Company designation for a premium molded grade graphite.

TABLE 2

Summary of "Intermetallic" Compositions Fabricated into Nibs for Testing as Experimental Die Materials

	Composition	Source	Fabrication
Metal Bonded	$ZrSiO_4$ - W	NMI	Hot Pressed
	TaC - W	Kennametal	Sintered
	Al_2O_3 - W	NMI	Hot Pressed
	ThO_2 - W	NMI	Hot Pressed
	Si_3N_4 - W	NMI	Hot Pressed
	* WC - Co	Kennametal	Sintered
	* TiC - Ni	Kennametal	Sintered
	* TaC - Ni	Kennametal	Sintered
	* TiC - Fe	Sintercast	Infiltrated
Self Bonded	TiB_2	Kaiser	Hot Pressed
	Al_2O_3	General Electric	Sintered
	ZrO_2	Zircoa	Sintered
	ZrC	Norton	Hot Pressed
	B_4C	Norton	Hot Pressed
Oxide Bonded	$MoSi_2$ - Al_2O_3	NMI	Hot Pressed
	$MoSi_2$ - ZrO_2	NMI	Hot Pressed
	$MoSi_2$ - ThO_2	NMI	Hot Pressed
	TiB_2 - Al_2O_3	NMI	Hot Pressed
	TiB_2 - ZrO_2	NMI	Hot Pressed
	TiB_2 - ThO_2	NMI	Hot Pressed
	ZrB_2 - Al_2O_3	NMI	Hot Pressed
	Si_3N_4 - MgO	NMI	Hot Pressed
Alloys	W - Ru	NMI	Cast, Wrought
	Ta - Ru	NMI	Cast, Wrought
	* W - Ta	Kennametal	Sintered
	* W - Re	Chase Brass	Sintered

* In process.



RF-9132

Fig. 1 - Hot pressed W - 33 $^{v}/o$ ThO_2 Tee nib (shrunk within steel casing).

W-ThO_2 was dry blended, and then hot pressed at 3200°F (1750°C) and one tsi for ten minutes in a preshaped ATJ grade graphite mold. (The spots on the nib are due to poor mixing which occurred because of agglomeration of the ThO_2.)

convenient than other forms of tungsten. The wool contains wire of various diameters from 1 to 10 mils. Consolidation of tungsten wool by isostatic pressing, cold rolling in a steel can and cold swaging in a steel tube was attempted, followed by cutting with a tool bit or an abrasive cut-off wheel.

Isostatic pressing in rubber under a unit pressure of about 90 tsi yielded a "bar" about one-inch diameter by approximately 1-1/2 inches long. Even with this pressure poor bonding was obtained, and the fibers did not hold together during the cutting operation. Isostatic pressing was therefore abandoned.

To consolidate the tungsten wool by canned rolling, the "picture-frame" method was used. An open cover "box" was fabricated by welding approximately one-inch high strips of steel around the periphery of a steel sheet, 4 inches by 8 inches by 1/8 inch thick. The tungsten wool was arranged carefully by hand in the box so that the orientation of the fibers favored the rolling direction. A cover plate was welded to the assembly, with a small hole left unwelded to permit air to escape during rolling. Unidirectional rolling was carried out at room temperature until the can ruptured. The total thickness of the composite at this point was approximately 5/16 inch, of which about 3/16 inch was tungsten. The ruptured steel covers were removed mechanically, and the resulting compacted tungsten wool was found to consist of densely matted lengths of kinked wire which were friable and readily separable by screening. Approximately three quarters of the fibers were less than one-half inch long, and some were as short as 0.005 inch. Approximately five pounds of usable fibers were obtained by the canned rolling method.

A third method -- cold swaging of steel-jacketed bundles of fibers -- gave the straightest and most uniform product. Tungsten wool was tamped into a steel tube one-inch diameter by 90 mil wall. The tube was cold swaged in a series of passes to about one-half inch diameter, at which point the steel casing ruptured at one spot, and swaging was discontinued. The individual tungsten wires were not reduced in cross-section, but they were packed densely by the swaging operation. The rod was then sectioned transversely into 0.125 inch lengths and the steel casing was consumed in nitric acid

solution. The compacted tungsten fibers were freed by tumbling on a screen with a few steel balls. Approximately three quarters of the fibers had a length of 0.125 inch, the cut length; some were shorter, and a small amount were longer (caused by doubling over). About two pounds of fibers fabricated by the swaging method have been processed completely, and an additional eight pounds have been tamped into steel tubes and are ready for swaging. The yield of usable fibers is roughly estimated to be 50 per cent -- in a three-foot long rod, approximately six inches at each end are not compacted sufficiently, and the remaining losses occur in the cutting operation. Higher yields can probably be obtained by swaging longer rods.

Since the swaging process produced fibers with the fewest kinks, the swaging process is the one that will be used for all future fiber manufacturing. A photograph of typical swaged fibers is given in Fig. 2.

2. Development of Internal Supports

To achieve economy in tooling costs and to assist in the solution to problems of supporting Tee dies externally, experiments were carried out to develop a successful internal support for round dies. Magnesia-stabilized zirconia (ZrO_2-MgO), a representative brittle material, was used as the prototype nib in these die support studies.

Three types of supports were tried, as shown in Fig. 3. A distinguishing feature of two of these supports (Figs. 3B and 3C) is a copper ring around the casing. Since the copper ring is slightly longer than the casing, the copper is compressed by the steel cone and isostatically supports the hardened steel casing; this design is a combination of the "soft" and "hard" supports investigated in past work.[2] The first (Fig. 3A) has no isostatic support (copper sleeve between casing and liner) and no nib cushion (copper ring between the casing and nib), the second (Fig. 3B) has an isostatic support and no cushion, and the third (Fig. 3C) has both support and cushion. Each ZrO_2 nib was shrunk into a steel casing with a 5-mil interference fit. A steel billet was extruded at a ram speed of 2.3 inches per second through each die at 2200°F (1200°C) and 80 tsi. The nib without isostatic support cracked severely; the others did not. In

6X RF-9094-2a

Fig. 2 - Tungsten fibers prepared by swaging and cutting.

The tungsten wool was loaded into a steel tube and swaged at room temperature until no further densification was observed. The rod was sectioned into short lengths and the steel shroud was removed in dilute nitric acid solution.

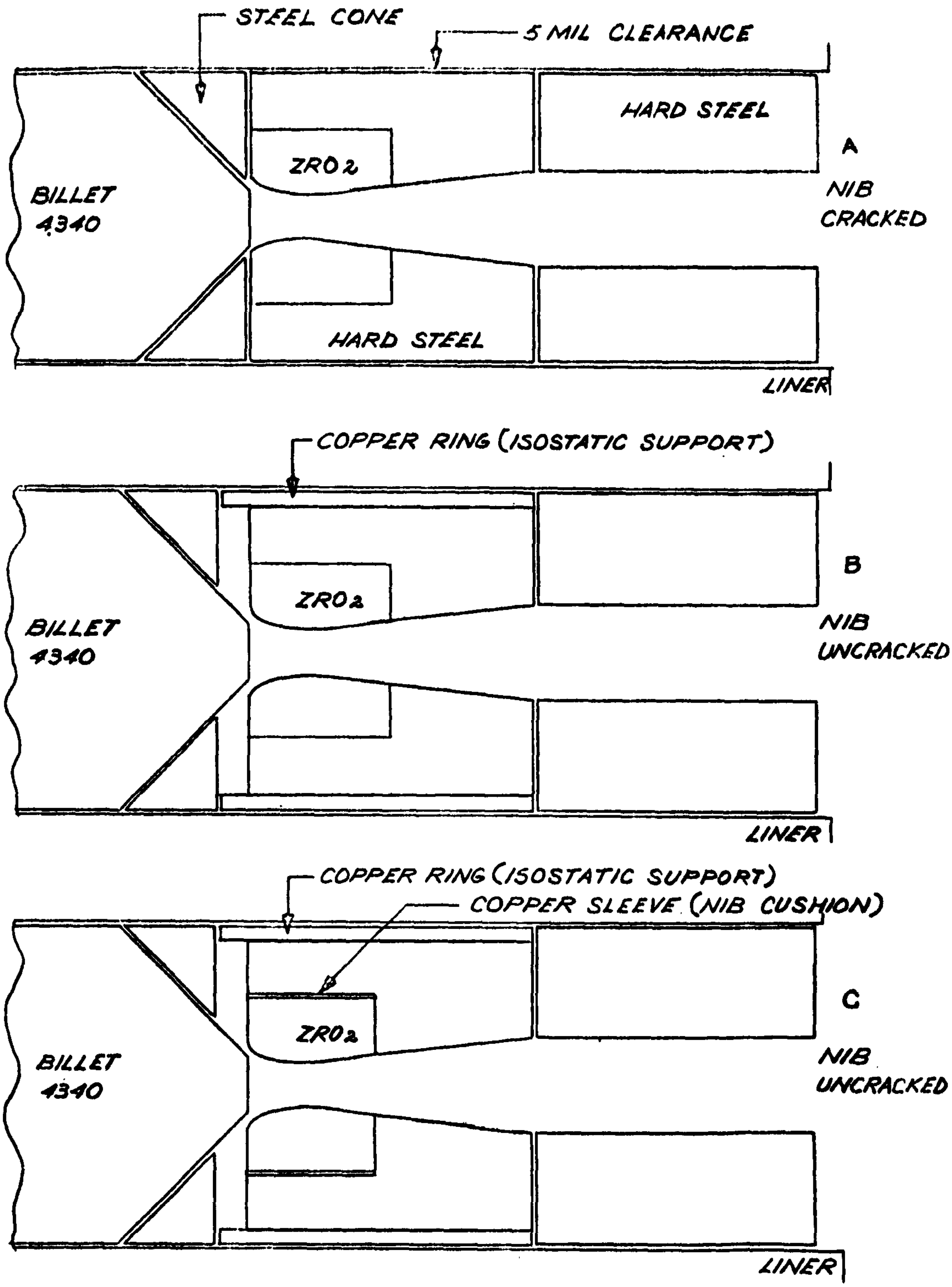


Fig. 3 - Designs of three internal supports. (All ribs shrunk into casings with 5-mil interference fit.) Drawing No. RA-2583.

A. Supported by liner.
B. Supported by copper ring (isostatic support).
C. Supported by copper ring with copper cushion.

fact, their performance indicates that massive ZrO_2 is a promising die material. Although the value of the nib cushion (copper ring adjacent to nib) is not demonstrated by these experiments, it is anticipated that a similar cushion made of aluminum can be dissolved in sodium hydroxide solution to free a used casing so that it can be reused with a new nib.

3. Evaluation of Substrates

To investigate substrates possibly superior to tool steel in utility and compatibility with prospective coatings, various materials were compared with 18-4-1 alloy steel hardened to 52 R_c and H21 steel hardened to 52 R_c, the latter coated with ZrO_2 by flame spraying. The experimental substrate materials were wrought tungsten, and stress relieved TZM and TZC molybdenum alloys. The experimental conditions were complicated by not having all die openings the same size (the materials were available from other work). The tungsten and the 18-4-1 alloy steel were tried in two die sizes, nominally 0.500 and 0.750 inch diameter, and a performance correlation is possible. Steel billets heated to 2200°F (1200°C) were extruded at a ram speed of 2.3 inches per second with glass lubrication (glass 1597B). Mica lube was used in the liner and a glass pad was used in the die. Up to five 4340 steel billets, each six inches long, were extruded through each die. The dies that did not fail in the extrusion of steel were used to extrude pure molybdenum at 3400°F (1870°C) with glass lubricant 1597A. The diameter of the die land was measured before and after each extrusion. The results of these measurements and comments on the condition of the dies are given in Table 3. From these data it can be concluded that under the conditions used, bare tungsten, TZM, and TZC are better die materials than bare tool steel, and that ZrO_2-coated tool steel is superior to the bare refractory metals tried. There was evidence, however, that after the sixth extrusion the ZrO_2 coating on the tool steel was about to spall because of severe cracking.

After the sixth extrusion the zirconia-coated steel die was sectioned to reveal the steel-ZrO_2 interface. The hardness of the steel 50 mils below

TABLE 3

Contrast in Die Performance as Revealed

by Successive Changing Diameters*

Die Material	Billets of AISI 4340 at 2200°F (1200°C)						Mo at 3400°F (1870°C)
	2.8 in. liner				3.5 in. liner		2.8 in. liner
	Sequential Extrusion Number						
	0	1	2	3	4	5	6
	Die land diameter in mils						
W	501	494	494	494	495	495	Severely reduced diameter
H21-ZrO_2	500	500	500	501	499	502	Near failure
18-4-1	507	506	505	513	Severe die wash	**	
TZC	747	747	747	731	754	729	Severely reduced diameter
TZM	707	709	709	704	731	677	" " "
W	750	751	750	747	743	742	" " "
18-4-1	753	753	750	743	Severe die wash	**	

* Billets extruded 2.3 in./sec., glass lubrication, graphite cutoffs, mica lube on liner, and glass pad on die.

** Failed in preceding extrusion.

the ZrO_2 was found to be 50 R_c. The thickness of the ZrO_2 was about 200 mils. The outstanding performance of this die may be attributable to the excessive thickness of the coating. If the coating had been about 30 mils, a standard thickness for coated dies, failure may have occurred earlier. Since some cracking was evident in the coated die after considerable use, a die made entirely of ZrO_2 may perform better than the ZrO_2-coated die because of the absence of the metal-oxide bonding problem and expansion coefficient differences, and low density in the sprayed ZrO_2 coating. The possible use of massive ZrO_2 as a die appears promising in view of its satisfactory performance in the die support study (Section B.2). Moreover, heavy ZrO_2 coatings may not be practical with Tee dies because of spalling that may be associated with a complicated shape.

4. Development of Testing Procedures

a. Indenter Test

An indenter test procedure conceived during the first quarter of the program[1] was intended to facilitate the testing of a large number of materials in a way more economical than actual extrusion tests of dies. The equipment was designed and fabricated in the first quarter, specimens prepared in the second quarter, and tested in the third quarter. Concurrently, coated dies were prepared with the same substrate and coating as the indenters. Tests were performed to determine whether a correlation in performance exists between specimens with the geometry of an indenter and with the geometry of a die. The results show that alignment of the indenter and billet are difficult to achieve and reproduce, a fact which prevents any useful comparison between the indenter results and the die results. The indenter test appears to contain greater complications than the extrusion die test without giving consistent or meaningful results. Consequently, further work with the indenter test facility is being postponed until a situation arises in the course of the program that indicates its resumption will be advantageous.

b. <u>Billet Heating and Loading Equipment</u>

To assure uniformity of conditions under which an experimental material is tested as a die, two existing furnaces have been modified to incorporate a special loading device.

The first furnace and loader, used for steel billets, is based on a design used by several contractors working on related programs. A billet, which has been coated with glass, is heated by 3000 cycle induction under flowing argon as it rests on a steel support. After the desired temperature is reached (12 min.) and maintained (1 min.), the billet is lowered on a hydraulic pedestal, automatically tipped horizontally, clasped with tongs manually, and placed on the press loader. The billet is automatically lifted to a position concentric with the liner cavity. The stem pushes it into the liner and through the die. The loading operation takes 20 seconds, and the extrusion less than one second.

The second furnace and loader, based on an original design, is used for molybdenum alloy TZM. The furnace is shown in Fig. 4. A billet which has been previously coated with glass is heated by induction in an argon atmosphere as it sits with its nose on a tantalum foil wafer resting on a carbon pedestal. The temperature is measured by an optical pyrometer, which can be used to sight into a drilled and tapped recess at the rear of the billet. (This recess is also used to load the billet into the furnace with a threaded rod.) After the extrusion temperature is reached (7 min.) and maintained (2 min.), a latch, which supports the pedestal, is released. The pedestal is pushed down and sideways by the weight of the billet, and the billet falls freely down a curved chute onto a platform behind the liner. The stem pushes the billet into the liner and through the die. The loading operation takes two to three seconds.

c. <u>Procedure for Evaluating Candidate Die Materials</u>

Another standardized procedure has been established for evaluating experimental die materials in view of the unsatisfactory results from the indenter test. A candidate material is tested as an internal round

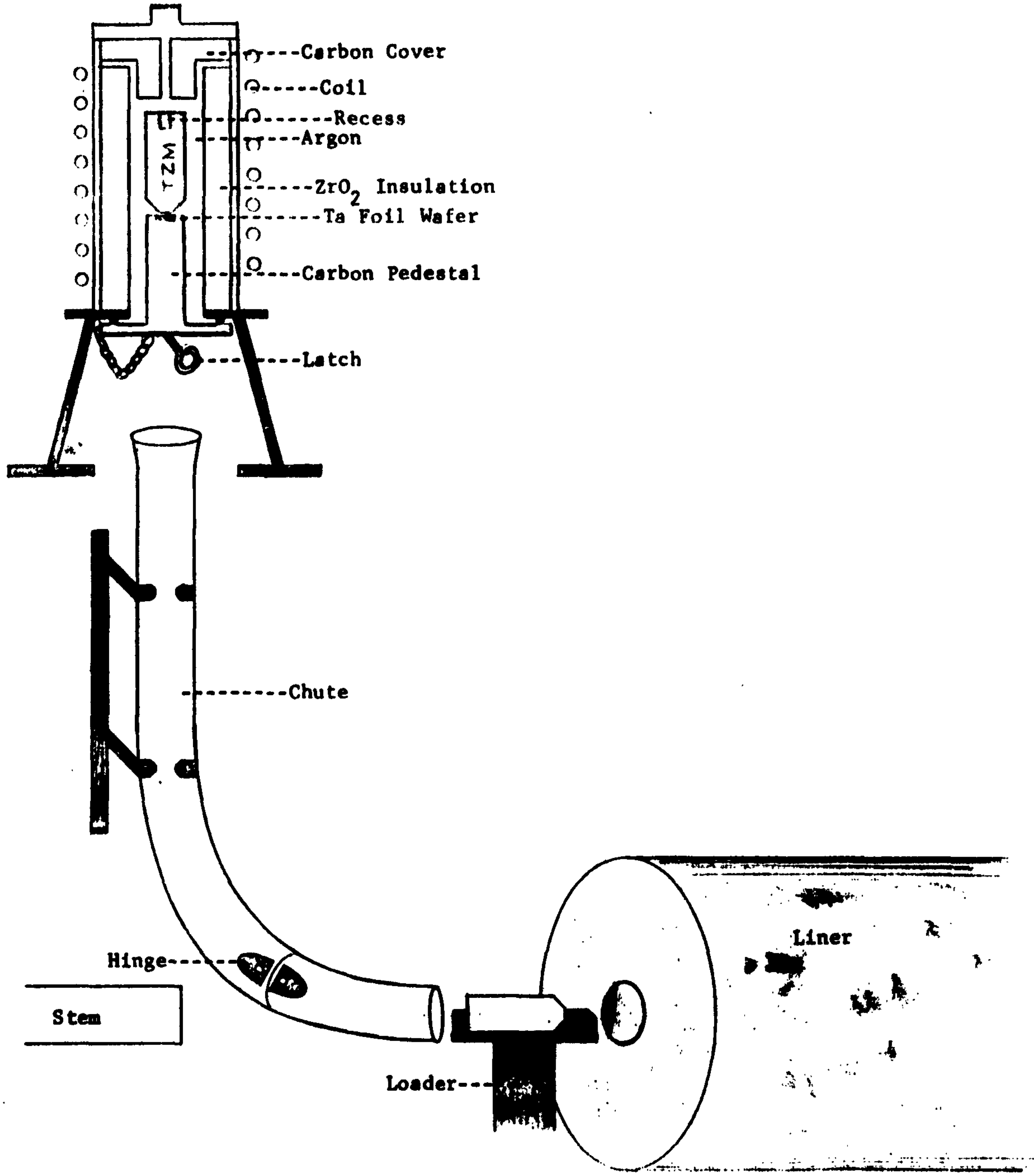


Fig. 4 - Sketch of high temperature furnace and loader. Drawing No. RA-2582.

Release of the latch causes the billet to free fall behind the liner from where it is pushed into the liner and through the die by the stem.

die for the extrusion of 4340 steel at 2200°F (1200°C) at a reduction ratio of 80X with 8-inch long billets. The die geometry is shown in Fig. 5. If performance is adequate, a second extrusion is carried out with the same die under the same conditions with a 12-inch long billet. If the material performs promisingly, a TZM billet is tried with a new die with the billet heated to 3400°F (1870°C) and at a reduction ratio of 22X. Die materials successful under these conditions will be fabricated into externally supported Tee's.

III. CONCLUSIONS

The status of the program at the end of the third quarter is as follows:

1. The fabrication of "intermetallic" round dies by hot pressing and grinding, and melting and forging has been successfully completed.

2. The fabrication of "intermetallic" Tees by hot pressing has been successfully demonstrated; however, some additional work on mold design is indicated.

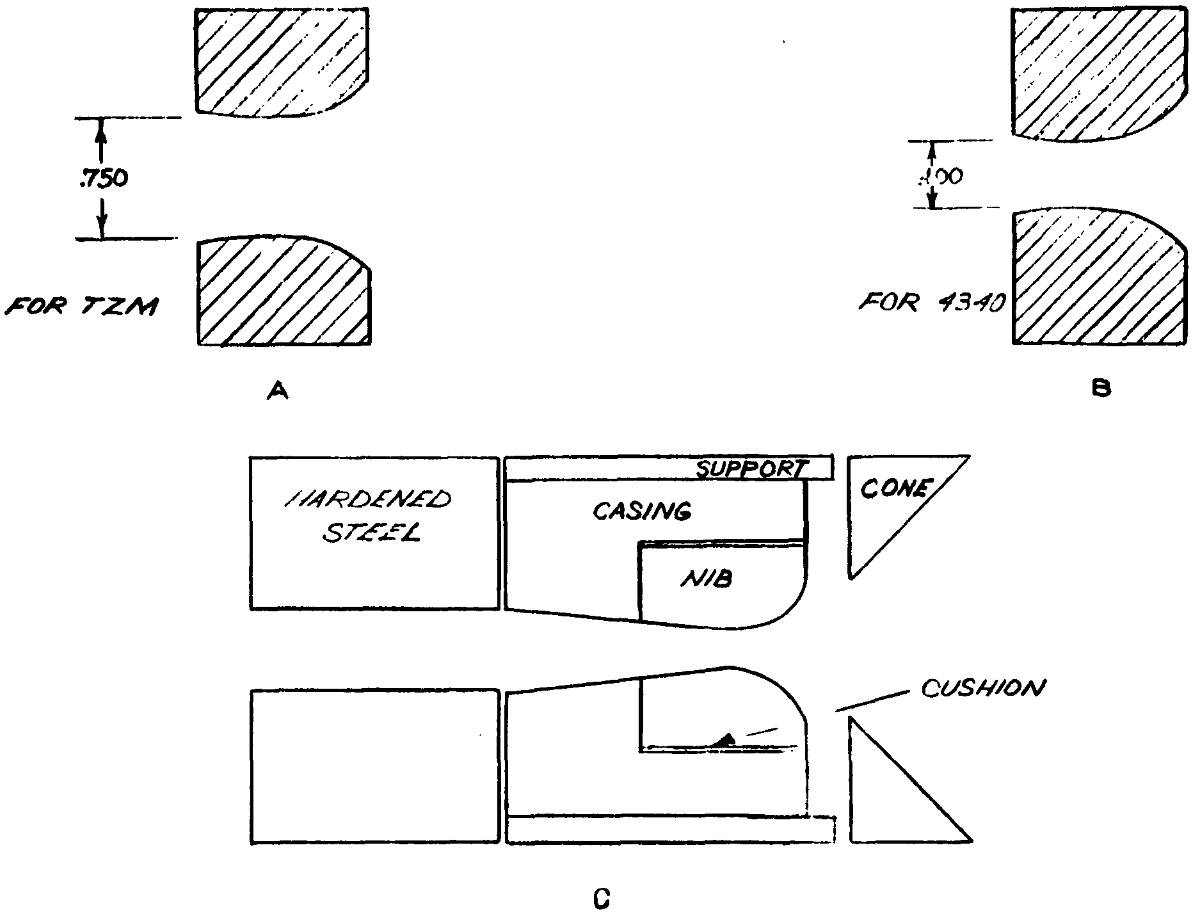
3. A simple process for the economic fabrication of tungsten fibers has been successfully accomplished. Reinforced ceramics can now be fabricated and tested.

4. Internal supports have been successfully developed to allow simpler and more economic testing of candidate materials. Information obtained from development of these supports will assist in the development of external Tee supports.

5. Wrought tungsten appears to offer promise as a substrate for coatings.

6. The evaluation procedure and tooling in the form of suitable furnaces and loading schemes have been formulated and tested.

7. The indenter test in its present state of development is not a satisfactory means of screening die materials. Further work with the indenter test will be held in abeyance.



<u>Fig. 5</u> - **Design of experimental dies. Drawing No. RA-2584.**

A. Geometry for TZM billets.
B. Geometry for 4340 steel billets.
C. Support design.

IV. FUTURE WORK

Experiments in the coming months will include the following: (1) testing of "intermetallic" rounds, (2) fabrication of "intermetallic" Tees and fiber reinforced rounds and Tees, (3) development of external supports for Tee dies, and (4) investigation of coatings on refractory metal substrates.

V. REFERENCES

1. Hunt, J. G., et al., Development of Dies for Extrusion of Complex Shapes of Steel and Refractory Alloys, First Interim Technical Progress Report to ASD for the Period June 15, 1962 through September 15, 1962, Nuclear Metals, Inc., West Concord, Mass., Report NMI-9700.3, December 17, 1962.

2. Hunt, J. G., et al., Development of Dies for Extrusion of Complex Shapes of Steel and Refractory Alloys, Second Interim Technical Progress Report to ASD for the Period September 16, 1962 through December 31, 1962, Nuclear Metals, Inc., West Concord, Mass., Report NMI-9700.6, March 4, 1963.

3. Kaufmann, A. R., et al., Refractory Metal Constitution Diagrams, Report to Wright Air Development Division, Nuclear Metals, Inc., West Concord, Mass., WADD Technical Report 60-132 (NMI-9216) June 1960.

4. Rapperport, E. J., and Geary, A. L., High Temperature Properties of Refractory Alloys, Summary Technical Report to the United States Atomic Energy Commission for the Period July 1, 1961 through June 30, 1962, Nuclear Metals, Inc., West Concord, Mass., USAEC Report NMI-1253, May 1963.

5. Tinklepaugh, J. R., "Metal Reinforcement and Cladding of Cermets and Ceramics," <u>Cermets</u>, Tinklepaugh, J. R., and Crandall, W. B., Editors, Reinhold Publishing Corp., New York, 1960, p. 170.

DISTRIBUTION

Copy No.

Distribution per list provided by Contractor: 1-143

Aeronautical Systems Division; Attn:

ASRCTB (5 cys)
ASRCMP-4, S. Inouye
ASW, Hazen A. Miles
ASRCMC, W. C. Ramke
ASYF, J. M. Troyan (2 cys)
ASRCE, J. Teres
ASNPFP, R. Farrington
ASRC, Dr. A. M. Lovelace
ASRCEM-1A, Mrs. N. Ragen

AFLC, WPAFB, Attn: MCPE: Col. R. O. Mitterling
FTD, WPAFB, Attn: TD-E2b
Watertown Arsenal, Attn: S. V. Arnold (2 cys)
Bureau of Naval Weapons, Attn: S. E. Samfilippo, AE 155
National Academy of Science, Attn: E. V. Bennett
Allegheny Ludlum Steel Corp., Watervliet, Attn: Extrusion Plant
Allegheny Ludlum Steel Corp., Brackenridge, Attn: R. K. Pitler
Aluminum Company of America, Attn: R. W. Andrews, Jr.
ASTIA, Arlington (20)
Bureau of Naval Weapons, Attn: Code PID-2 (2)
DTI, Oak Ridge, Attn: Hugh Voress
AFSC, Andrews AFB, Attn: SCR-2, C. W. Kniffin
NASA, Attn: George Mandel
Ajax Magnathermic Corp., Attn: J. A. Logan
Hubert Altwicker, Lebanon, Ohio
Armour Research Foundation, Attn: Frank Crosely
AVCO Mfg., Co., Attn: W. H. Panke
Baldwin-Lima-Hamilton Corp., Philadelphia, Attn: Dr. F. J. Kent
Battelle Memorial Inst., DMIC, Ohio
Beech Aircraft Corp., Wichita, Attn: Emmet Utter
Bell Helicopter Co., Ft. Worth, Attn: Neil J. McKenzie
Boeing Co., Aero Space Div., Seattle, Attn: George Hughes
William L. Bruckart, Pittsburgh
Canton Drop Forging and Mfg. Co., Attn: Chandis Brauchler
Atlantic Research Corp., Alexandria, Virginia
AVCO Corp., R and Adv. Dev. Div., Attn: John V. Erickson
Babcock and Wilcox Co., Beaver Falls, Pa., Attn: James Barrett
Barogenics, Inc., NYC, Attn: Alexander Zeitlin
Battelle Memorial Inst., Attn: A. M. Sabroff
Bell Aerospace Corp., Buffalo, Attn: H. A. Campbell
Bendix Products Div., South Bend, Attn: David M. Scruggs
The Boeing Co., Wichita, Attn: Walter Burnham
California Inst. of Tech., JPL, Attn: I. W. Newlan
General Dynamics Corp./Convair, San Diego, Attn: J. H. Famme
General Dynamics Corp./Astronaut, San Diego, Attn: V. G. Mellquist
James W. Conrad, Pittsburgh
Crucible Steel Co. of America, Pittsburgh, Attn: Dr. Walter Finlay

DISTRIBUTION (continued)

Copy No.

Curtiss Division, Caldwell, New Jersey, Attn: W. C. Schulte
Douglas Aircraft Co., Inc., Long Beach, Cal., Attn: C. B. Perry, C-345
E. I. duPont deNemours and Co., Wilmington, Attn: Dr. E. M. Mahla
Fansteel Metallurgical Corp., N. Chicago, Attn: Dr. A. B. Michael
General Electric Co., Cincinnati, Attn: G. J. Wile
Climax Molybdenum Co., Detroit
Climax Molybdenum Corp., NYC, Attn: Dr. Janice Briggs
Corning Glass Works, N.Y.
Wright Aeronautical Division, Wood-Ridge, N.J., Attn: Jesse Sohn
Metals Processing Div., Buffalo, Attn: A. Roubloff
Douglas Aircraft Co., Inc., Santa Monica, Attn: A. J. Carah
Dow Chemical Co., Midland, Mich., Attn: Dr. T. E. Leontis
Erie Foundry Co., Attn: J. E. Wilson
The Garrett Corp., Los Angeles
General Electric Co., Cleveland, Attn: J. H. Keeler
Harvey Aluminum, Inc., Torrance, Cal., Attn: G. A. Maudry
Hughes Tool Company, Culver City
Jones and Laughlin Steel Corp., Pittsburgh, Attn: Robert S. Orr
Kawecki Chemical Co., Attn: Edwin V. Bielecki
Ling-Temco-Vought, Inc., Dallas, Attn: Library 1-63101
Lockheed Aircraft Corp., Marietta, Ga., Attn: Roy A. MacKenzie
The Martin Co., Denver, Attn: R. F. Breyer
McDonnell Aircraft Corp., St. Louis, Attn: R. D. Detrich
NORAIR Div., Hawthorne, Cal., Attn: J. A. Van Hammersveld
Gruman Aircraft Eng'g., Corp., Long Island
H. M. Harper Co., Attn: E. A. Channer
Haynes Stellite Co., Kokomo
Hunter Douglas Div., Riverside, Cal.
Kaiser Aluminum and Chem. Corp., Dayton
Lockheed Aircraft Corp., Burbank, Attn: Elliot Green
Lockheed Aircraft Corp., Sunnyvale, Attn: Max Tatman
Lombard Corp., Youngstown
Martin Marietta Corp., Attn: George C. Pfaff, Jr.
Martin Company, Baltimore, Attn: Science-Tech. Library
New England Materials Lab., Inc., Bedford
North American Aviation, Inc., Los Angeles
North American Aviation, Inc., Los Angeles, Attn: Walter Rhineschild
P. R. Mallory and Co., Inc., Attn: A. S. Doty
Rensselaer Polytechnical Inst.
Republic Steel Corp., Cleveland, Attn: R. W. Kollar
Revere Copper and Brass Co., Inc., NYC., Attn: C. J. Plovanich
Ryan Aeronautical Co., San Diego, Attn: Engineering Library
Solar Aircraft Co., San Diego, Attn: F. M. West
Stanford Research Inst.
Steel Improvement and Forge Co., Cleveland
Thompson-Ramo-Woolridge, Inc., Cleveland, Attn: Dr. A. S. Nemy
North American Aviation, Inc., Columbus, Attn: Plant Engineering Lib.
Oregon Metallurgical Corp., Albany, Attn: Frank Vandenburgh
Reactive Metals, Inc., Niles, O., Attn: George W. Cleveland

Copy No.

Republic Aviation Corp., Long Island, Attn: A. Kastelowitz
Reynolds Metals Co., Washington, D. C., Attn: Stuart Smith
Rohr Aircraft Corp., Chula Vista, Attn: F. E. Zimmerman
Sandia Corp., Livermore, Attn: M. W. Mote, Jr.
Southern Research Inst., Birmingham, Attn: A. C. Willhelm
Stauffer Metals Co., Richmond, Cal.
Superior Tube Co., Norristown, Pa.
United Aircraft Corp., E. Hartford
United Aircraft Corp., Bridgeport
Universal Cyclops Steel Corp., Bridgeville, Pa., Attn: Charles Mueller
Wah Chang Corp., Albany, Oregon, Attn: Mabel E. Russell
Wyman-Gordon Co., N. Grafton, Mass., Attn: Arnold Rustay
Wolverine Tube, Allen Park, Mich., Attn: F. C. Eddens
United States Steel Corp., Pittsburgh
Vanadium Corp. of America, NYC, Attn: C. N. Cosman
Wah Chang Corp., NYC, Attn: K. C. Li, Jr.
Westinghouse Electric Corp., Blairsville, Pa., Attn: F. L. Orrell
Oak Ridge National Laboratory, Attn: W. C. Thurber
Kaiser Aluminum and Chem. Corp., Permanente, Cal., Attn: R. Mogstad
Aerospace Ind. Association of America, Inc., Washington, D. C., Attn: S. D. Daniels
General Electric Co., Philadelphia, Attn: C. M. Zvanut
General Electric Co., Cleveland, Attn: Stephen Foldes

Internal:

J. L. Klein	144
P. Loewenstein	145
A. M. White	146
R. G. Jenkins	147
J. G. Hunt	148
S. Abkowitz	149
Document Library	150-151

Aeronautical Systems Division
Wright-Patterson Air Force Base, Ohio
Report No. ASD-TDR 7-946 (III)
DEVELOPMENT OF DIES FOR EXTRUSION OF COMPLEX SHAPES OF STEEL AND REFRACTORY ALLOYS.
Interim report, May 63, 20p. incl illus., tables, refs.

Unclassified Report

Work continued on methods of fabricating and evaluating dies suitable for the high temperature extrusion of steel and refractory metals of complex shapes. Fabrication of ceramic and "intermetallic" dies by hot pressing and by melting and forging continued. Successful methods of producing tungsten fibers for reinforced dies were developed. A die support system based on the concept of hydrostatic support for the die casing has been developed and shown to be successful in preventing the cracking of brittle die nibs during extrusion.

1. Materials - Processing
2. Dies-Forming
3. Dies-Materials
4. Steel-Extrusion
5. TZM-Extrusion
6. Extrusion of Tees

I. ASD Proj. 7-946
II. Contract AF 33(657)-8798
III. Nuclear Metals, Inc., West Concord, Mass.
IV. J. G. Hunt, et al.
V. Secondary Report No. NMI-9700.9
VI. In ASTIA Collection.

Aeronautical Systems Division
Wright-Patterson Air Force Base, Ohio
Report No. ASD-TDR 7-946 (III)
DEVELOPMENT OF DIES FOR EXTRUSION OF COMPLEX SHAPES OF STEEL AND REFRACTORY ALLOYS.
Interim report, May 63, 20p. incl illus., tables, refs.

Unclassified Report

Work continued on methods of fabricating and evaluating dies suitable for the high temperature extrusion of steel and refractory metals of complex shapes. Fabrication of ceramic and "intermetallic" dies by hot pressing and by melting and forging continued. Successful methods of producing tungsten fibers for reinforced dies were developed. A die support system based on the concept of hydrostatic support for the die casing has been developed and shown to be successful in preventing the cracking of brittle die nibs during extrusion.

1. Materials - Processing
2. Dies-Forming
3. Dies-Materials
4. Steel-Extrusion
5. TZM-Extrusion
6. Extrusion of Tees

I. ASD Proj. 7-946
II. Contract AF 33(657)-8798
III. Nuclear Metals, Inc., West Concord, Mass.
IV. J. G. Hunt, et al.
V. Secondary Report No. NMI-9700.9
VI. In ASTIA Collection.